Gas breakdown in an atmospheric pressure radio-frequency capacitive plasma source

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Gas breakdown is studied in an atmospheric pressure rf capacitive plasma source developed for materials applications. At a rf frequency of 13.56 MHz, breakdown voltage is largely a function of the product of the pressure and the discharge gap spacing, approximating the Paschen curve. However, breakdown voltage varies substantially with rf frequency due to a change in the electron loss mechanism. A large increase in breakdown voltage is observed when argon, oxygen, or nitrogen is added to helium despite their lower ionization potential. Discussion is given for optimal breakdown conditions at atmospheric pressure. © *2001 American Institute of Physics.*
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I. INTRODUCTION

Materials processing using plasmas is a vital industrial technology in many areas including electronic, aerospace, automotive, biomedical, and toxic waste management industries. This is because of the unparalleled capability of plasmas for production of chemically active species at low gas temperature while maintaining high uniform reaction rate over relatively large areas.[1,2] Currently, the majority of plasma processing is done at low pressures and the vacuum operation is viewed as a necessary requirement. In principle, however, atmospheric pressure plasmas can provide a critical advantage over the widely used low pressure plasmas (e.g., magnetron, reactive ion etchers, inductively coupled plasmas, etc.), as they do not require expensive and complicated vacuum systems. Without a vacuum system, the cost of materials processing could be reduced substantially and issues related to vacuum compatibility of materials would be mitigated. Therefore, the use of atmospheric pressure plasmas could greatly expand the current scope of materials processing using plasmas. This has prompted recent development of a number of novel atmospheric pressure plasma sources including the atmospheric pressure plasma jet (APPJ),[3–7] the cold plasma torch,[8] the one atmosphere uniform glow discharge plasma,[9] microhollow cathode discharge,[10] and surface-wave discharge.[11]

For example, the APPJ operates in a capacitively coupled configuration using rf power and produces a stable, oscillating steady state discharge without dielectric material between the electrodes. Importantly, the discharge fills the volume between the electrodes and is free of the filaments, streamers, and arcing that are often observed in other atmospheric pressure plasma sources. The gas temperature in the discharge is typically between 50 and 300 °C. Thus, thermal damage to treated materials can be easily avoided. The primary feedgas of the APPJ is helium, to which a small fraction (0.5%–3%) of reactive gases (e.g., oxygen and/or car-

bon tetrafluoride, water vapor) is added to generate a flux of chemically active species. These reactive species are transported with the gas flow out of the discharge and impinge on the surface downstream. In this way, the surface to be treated does not require immersion inside the source and is not directly exposed to the plasma. To date, the APPJ has been used to etch polyimide, tungsten, tantalum, and silicon dioxide, and to deposit silicon dioxide films at rates comparable to those achieved in low pressure discharge systems.[4,5] We have also demonstrated the potential use of the APPJ for decontamination of chemical and biological warfare (CBW) agents by destroying surrogates of mustard blister agent, VX nerve agent, and Anthrax spores.[6]

Though promising, various issues including discharge stability, gas and surface phase chemistry, chemical reactivity and protection from thermal damage, need to be addressed for practical uses of these novel atmospheric pressure plasma sources. In particular, the gas breakdown at atmospheric pressure often results in a streamer or a filamentary arc instead of a stable glow-like discharge. This is because gas breakdown at atmospheric pressure generally requires a much higher voltage, in excess of 1 kV, compared to low pressure breakdown. The high breakdown voltage causes a rapid multiplication of electrons after breakdown, resulting in a streamer or a filamentary arc, as in the case of corona discharges and dielectric barrier discharges. Therefore, if one can lower the breakdown voltage, it becomes easier to manage the issue of discharge stability at atmospheric pressure.

In this article, we present and analyze the gas breakdown in the APPJ which operates in a capacitively coupled configuration using radio frequency power, typically at 13.56 MHz. This is followed with a discussion on optimal breakdown conditions to obtain stable glow-like plasmas at atmospheric pressure that are attractive for materials applications.

II. EXPERIMENTS

As shown in Fig. 1, experiments were performed using two planar square electrodes: a rf powered top electrode and

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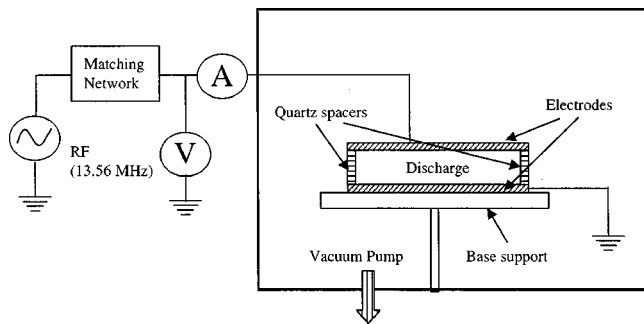
FIG. 1. Schematic of the experimental setup. The discharge system is placed in a vacuum chamber to reduce impurities in the discharge and to operate at the desired pressure, up to atmospheric pressure.

FIG. 3. Breakdown voltage measurements as a function of rf frequency in pure helium for a gap spacing of 0.16 cm and a gas pressure of 600 Torr.

a grounded bottom electrode. The surface area of the electrodes was 100 cm^2 and the gap spacing between the electrodes was varied from 0.1 to 0.97 cm using quartz spacers. To minimize the impurities in the discharge, the discharge assembly was placed in a vacuum chamber, pumped by a 500 1/s turbopump. The base pressure of the vacuum chamber was 3×10^{-7} Torr. During the experiments, the chamber was pumped out below 1×10^{-3} Torr and filled with a high purity gas (99.9995% or better for helium, oxygen, and argon, and 99.95% or better for nitrogen) mixture at room temperature and at pressures ranging from 10 to 600 Torr prior to the measurements. The atmospheric pressure at the altitude of Los Alamos is about 590 Torr, which limited the operation to below 600 Torr to prevent overpressure in the vacuum chamber. The rms value of the breakdown voltage was measured using a high voltage probe (Tektronix P6015A with a bandwidth of 75 MHz) and were recorded on a digital oscilloscope (Tektronix TDS 640 with a 2 G sample/s sampling rate and a bandwidth of 500 MHz). At 13.56 MHz, a tuned impedance probe (Advanced Energy rfZ 60 probe) was also used to measure the breakdown voltage and the agreement between the two measurements was reasonable (within 15%).

Figure 2 shows the breakdown voltage measurements in pure helium at 13.56 MHz as a function of the product of the gas pressure and the electrode gap spacing, pd. The measure-

ments were made using the high voltage probe and consist of breakdown voltages for five different gap spacings with varying gas pressure. It is noted that the breakdown voltage varies about 5% for day-to-day measurements, possibly due to changes in the surface condition of the electrodes. However, the breakdown voltages were reproducible to within ± 3 V for the present experiments conducted during a relatively short period of time (~ 2 h). The breakdown voltage measurements are shown for pd values only between about 10 and 200 Torr cm. This is because the discharge becomes unstable and turns into a filamentary arc immediately after breakdown for pd values above 240–270 Torr cm. When the pd value is reduced between 10 and 20 Torr cm, the breakdown voltage starts increasing with decreasing pd values, seen in the breakdown voltage measurement for the gap spacing of 0.18 cm. At the same time, the breakdown takes place between the outer edge of the rf electrode and the vacuum chamber wall rather than between the electrodes. For this reason, we could not measure the breakdown voltage below 10 Torr cm and the data point for the lowest pd value represents the minimum breakdown voltage for that given gap spacing.

As shown in Fig. 2, the breakdown voltage is largely a function of pd and the minimum breakdown voltage is obtained between 10 and 20 Torr cm. However, the minimum breakdown voltage tends to be lower for larger gaps, e.g., 62 V for the 0.1 cm of gap spacing and 150 Torr gas pressure, but 49 V for 0.25 cm and 75 Torr, and 37 V for 0.97 cm and 10 Torr. In addition, the breakdown voltage for the 0.97 cm gap spacing shows a relatively large discrepancy from those for the smaller gaps with the same pd value. These results indicate that the breakdown voltage obeys the Paschen's law approximately though small discrepancies are seen beyond the experimental uncertainty. On the other hand, little variation, less than 10%, was observed in breakdown voltages for various electrode materials, e.g., aluminum, copper, molybdenum, and stainless steel.

In Fig. 3, the breakdown voltage in pure helium is shown as a function of rf frequency. The measurements were made with a 0.16 cm electrode gap and 600 Torr gas pressure. At frequencies above 8 MHz, the breakdown voltage decreases monotonically but only slightly, from 149 V at 10 MHz to
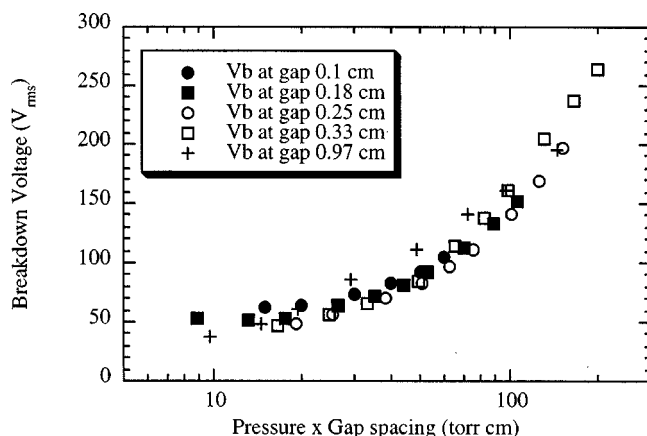
FIG. 2. Breakdown voltage measurements as a function of gas pressure and gap spacing in pure helium using aluminum electrodes. Five different gap spacings are used, 0.1, 0.18, 0.25, 0.33, and 0.97 cm.
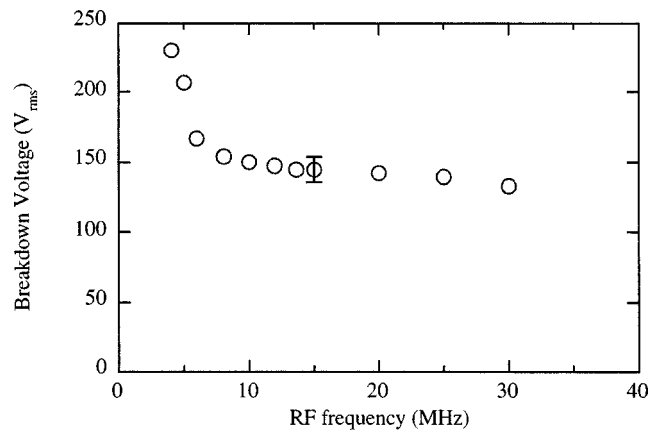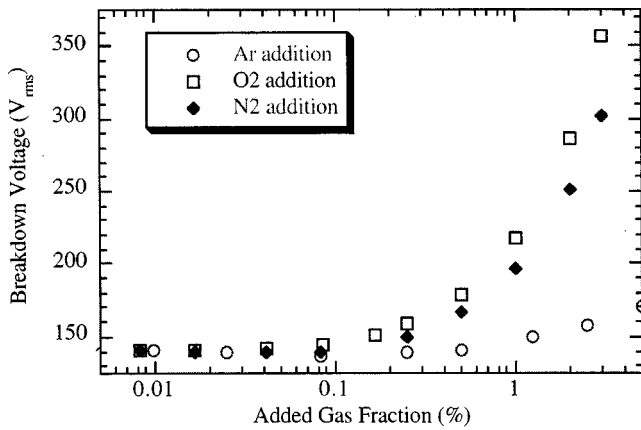
FIG. 4. Breakdown voltage measurements as a function of gas composition with addition of argon, nitrogen, and oxygen to pure helium. Other discharge parameters are: gap spacing $= 0.16$ cm, gas pressure $= 600$ Torr, and rf frequency $= 13.56$ MHz.

133 V at 30 MHz. In comparison, the breakdown voltage increases rapidly with decreasing frequency below 8 MHz, from 154 V at 8 MHz to 230 V at 4 MHz. Below 4 MHz, the discharge becomes unstable and turns into a filamentary arc immediately after breakdown.

The breakdown voltage varies substantially with the addition of other gases, as shown in Fig. 4. With addition of argon, the breakdown voltage initially decreases by a small, but finite amount, from 142 V in pure helium to 137 V with the addition of 0.08% of argon. Further addition of argon, however, increases the breakdown voltage substantially, up to 170 V for 5% argon in the gas mixture. In comparison, addition of nitrogen or oxygen yields no discernable initial decrease in the breakdown voltage and the breakdown voltage increases very rapidly when a substantial fraction of oxygen or nitrogen is added to the helium. For example, the breakdown voltage increases to 357 V for a 3% oxygen mixture and to 302 V for a 3% nitrogen mixture. Above 4%, the discharge turns into a filamentary arc for both oxygen and nitrogen.

III. DISCUSSION

The gas breakdown by high-frequency (HF) electric fields has been studied in detail by Brown and our analysis will be based on his theory.[12] According to Brown, the gas breakdown by HF fields occurs when the volume ionization equals the electron loss by diffusion. Based on this diffusion theory, he derived the conditions for HF breakdown similar to the Paschen curve for dc breakdown as breakdown phenomena is described by a set of three independent variables. One set of these variables is $E\Lambda$, $p\Lambda$, and $p\lambda$, where E is the breakdown electric field, Λ is the characteristic diffusion length, p is the gas pressure, and λ is the wavelength for HF field. For the parallel plate geometry used in this study, Λ is given as d/π and E is given as V_b/d, where V_b is the breakdown voltage. In addition, he showed that gas breakdown at high gas pressure can be described by only two variables, $E\Lambda$ and $p\Lambda$, independent of $p\lambda$ as long as the diffusion is the dominant electron loss mechanism. This result agrees

with the breakdown voltage measurement result in Fig. 2. Furthermore, the diffusion theory indicates that the secondary electron emission from the electrodes plays a small role in HF breakdown and the breakdown voltage does not depend on the electrode materials.

However, the validity of the diffusion theory requires the electron loss to be diffusion dominated and this condition requires a close examination in the present experiments. To estimate the electron loss to the electrodes during the gas breakdown, the motion of electron fluid is described as a sum of drift motion and diffusion,[13] as shown in

$$n_e v_e = -n_e \mu_e E_b - D_e \nabla n_e, \qquad (1)$$

where E_b is the vacuum electric field at breakdown, μ_e is the electron mobility, and D_e is the electron diffusivity. The use of a fluid description for electron motion should be reasonable at atmospheric pressure due to the very frequency collisions between electrons and neutrals. The *rhs* of Eq. (1) can be rewritten using the Nernst–Townsend relation and the characteristic energy of the electrons,[14] as in

$$
\begin{aligned}
n_e v_e &= -n_e \mu_e \left(E_b + \frac{D_e}{\mu_e} \frac{\nabla n_e}{n_e} \right) \\
&= -n_e \mu_e \left(E_b + \epsilon_e \frac{\nabla n_e}{n_e} \right) \\
\Rightarrow v_e &= \frac{dx_e}{dt} = -1.414 \mu_e \frac{V_b}{d} \cos(\omega t) - \mu_e \epsilon_e \frac{\nabla n_e}{n_e},
\end{aligned}
$$
$$(2)$$

where V_b is the rms breakdown voltage, d is the gap spacing, ω is the angular frequency of the electric field, and ϵ_e is the characteristic energy of the electrons, which is given a function of the reduced electric field, E/p.[15–18]

Each term in the rhs of Eq. (2) can be estimated using the experimental measurements of the breakdown voltage in Figs. 2 and 3. For breakdown of the 0.18 cm gap at 13.56 MHz, the electron characteristic energy in helium is about 1.8 eV at the breakdown electric field of 844 V(rms)/cm. Since the length scale for the electron density gradient is on the order of $d/2$, the drift motion dominates the diffusion by a factor of $60 (= 1.414 V_b/2\epsilon_e)$. However, this oscillatory drift motion produces no net displacement for the electrons, thus no electron loss to the electrodes when integrated over a rf period, as long as the amplitude of this electron oscillation is much smaller compared to the gap spacing between the electrodes. This limit on the amplitude of electron oscillation was discussed by Brown and needs to be satisfied for the diffusion theory to be valid. In the case of large electron oscillation amplitude, comparable to or larger than a gap spacing, the electron loss is controlled by the fast drift motion and the breakdown voltage increases from its value given as by the diffusion theory.

To estimate the amplitude of the electron oscillation, Eq. (2) is solved approximately by ignoring the diffusion term, as shown in

$$\frac{dx_e}{dt} \approx -1.414 \mu_e \frac{V_b}{d} \cos(\omega t) \Rightarrow A \approx 1.414 \langle \mu_e \rangle \frac{V_b}{\omega d}, \qquad (3)$$

where A is the amplitude of electron oscillation and $\langle\mu_e\rangle$ is the average electron mobility over the time varying electric field strength. For our qualitative discussion, we use a constant for the product of the pressure and the mobility $p\langle\mu_e\rangle$, 9×10^5 Torr cm^2/(V s) in the following calculation. This is because the mobility is roughly inversely proportional to the gas pressure for the reduced field between 1 and 10 V/(cm Torr) that are relevant to the current experiments.[15,18] Then, from Eq. (3), the electron oscillation amplitude is 0.021 cm for the gas breakdown of the 0.18 cm gap at 13.56 MHz. This result indicates that the diffusion theory may apply to the present experimental conditions with a reasonable accuracy, thus explaining the experimental result in Fig. 2 that the breakdown voltage is largely a function of pd, independent of electrode gap spacing.

However, the electron oscillation amplitude is not very small compared to the gap spacing, thus some discrepancies from the HF Paschen curve may occur, particularly for smaller gap spacings, lower gas pressures (via increasing electron mobility), and lower rf frequencies. For example, the electron oscillation amplitude is 0.062 cm and is more than half of the gap spacing, for the minimum breakdown (62 V) of 0.1 cm gap spacing at 150 Torr. On the other hand, the electron oscillation amplitudes are smaller fractions of the gap spacing for larger gaps, 0.039 cm for the minimum breakdown (49 V) of 0.25 cm gap at 75 Torr and 0.057 cm for the minimum breakdown (37 V) of 0.97 cm gap at 10 Torr. This result is consistent with the observed decrease in the minimum breakdown voltage for the larger gaps in Fig. 2, since the effect of electron drift loss decreases with increase in gap spacing.

Equation (3) can also be used to explain the observed increase of breakdown voltage with a decrease in rf frequency, as shown in Fig. 3. This is because the electron oscillation amplitude increases to a substantial fraction of the gap spacing with decreasing rf frequency. For the 0.16 cm gap at 600 Torr, the electron oscillation amplitude would increase from 0.0094 cm at 30 MHz to 0.035 cm at 8 MHz, and to 0.070 cm at 4 MHz using a constant breakdown voltage of 133 V measured at 30 MHz. This result agrees with the experiments, because the electron drift loss becomes significant only when the oscillation amplitude reaches a substantial fraction of the gap spacing, e.g., below 8 MHz. Similar results have been obtained using neon, nitrogen, and hydrogen at lower gas pressures.[19]

As shown in Fig. 4, the breakdown voltage is a strong function of the gas composition. For argon addition to helium, the breakdown voltage initially decreases by a small but finite amount (5 V from 142 V) with the addition of 0.08% of argon. This decrease in breakdown voltage can be attributed to Penning ionization of argon atoms by helium metastables. The work by Penning and Addink shows that a large reduction in breakdown voltage, a factor somewhat greater than 4, can be obtained when 0.03% of argon is added to neon for dc breakdown.[20] Interestingly, the decrease in breakdown voltage due to the Penning effect in the present experiment is rather small. A possible explanation is that the efficiency of gas breakdown increases for rf fields compared to dc fields. For example, the breakdown voltage is about

145 V rms at 100 Torr cm (0.16 cm gap at 600 Torr) in our system using rf fields, while the dc breakdown voltage in helium is about 800 V for the same pd value.[21] This high efficiency of gas breakdown using rf fields may mask the effect of Penning ionization.

After an initial decrease due to Penning ionization, the breakdown voltage increases with further addition of argon. This can be understood by noting that in a mixture of two gases the breakdown voltage often lies between those of the constituents.[22] The breakdown voltage for argon for high pd values, above 10 Torr cm, is higher than for helium both for dc and HF fields.[12,16] Also, it is noted that the Townsend first ionization coefficient is larger for helium than for argon for reduced fields (electric field strength divided by gas number density) below 10^{-15} V cm^2. Since the typical reduced field in our system is about 10^{-16} V cm^2 or less, the volumetric ionization rate will be smaller for argon than for helium. This increase in breakdown voltage for argon may be explained by the fact that the argon atom has many more excited states than the helium atom. Thus, electrons can lose their energy more effectively in argon before ionizing argon atoms. This loss of electron energy, in particular for the high energy electrons which are mostly responsible for ionization, will decrease the rate of ionization in the system. On the other hand, the charged particle loss rate will not vary much since the motion of the electrons will be largely determined by the collisions with the majority species, i.e., helium. Thus, the breakdown voltage will increase with the addition of argon to maintain particle balance.

A similar explanation can be given for the increase in breakdown voltage with addition of oxygen and nitrogen in the discharge. For a given fractional gas addition, however, a greater increase in breakdown voltage for molecular gases is due to additional electron energy loss channels, such as vibrationally and rotationally excited states and the molecular dissociation. For dc breakdown, increases in breakdown voltage with addition of oxygen and nitrogen have been reported, by a factor of 2 with 5% addition of oxygen[22] and by a factor of 5.5 with 20% addition of nitrogen.[22] In addition, the large electron affinity of oxygen molecules and the resulting electron loss by electron attachment may explain the larger increase of breakdown voltage for oxygen addition than that for nitrogen addition.

These results show that theoretically a small gap spacing, a high rf frequency, and a gas mixture consisting mostly of helium with a small addition of the reactive species is needed to obtain low breakdown voltage and to produce a stable capacitive discharge at atmospheric pressure. In practice, however, it is desirable to use as large a gap spacing and as low a rf frequency, as possible. This is because a large gap increases the area where the reactive radicals produced in the discharge react with materials and the cost of power supply and its efficiency favors a low rf frequency. In addition, the plasma source would produce a large amount of reactive radicals if a large amount of reactive species, including electronegative gases like oxygen and fluorine, can be used in the discharge though the optimum amount may depend on details of gas and surface chemistry. These conflicting requirements bring a question of an optimum breakdown condition

for an efficient operation of a practical plasma source.

As for the optimum gap spacing, the breakdown voltage is largely a function of pd independent of the gap spacing, as shown in Fig. 2. This is valid as long as diffusion is the dominant electron loss mechanism and additional electron loss such as drift loss will increase the breakdown voltage from its value given in Fig. 2 for the same pd. In addition, as mentioned earlier, the discharge becomes unstable and turns into a filamentary arc immediately after breakdown for pd values above 240–270 Torr cm. This sets the maximum gap spacing of about 0.4 cm, corresponding to a pd of 240 Torr cm, in which a stable glow-like discharge can be produced at atmospheric pressure for 13.56 MHz. As for the optimum rf frequency, one can use a higher frequency to reduce the drift loss of electrons and to lower the breakdown voltage, compared to dc or lower frequencies. However, the advantage of increasing the frequency diminishes once the electron loss is dominated by diffusion, as shown in Fig. 3. Moreover, the use of a very high frequency is not practical, as the power supply need to provide a very large displacement current in the gap prior to the breakdown. Therefore, we estimate the optimum rf frequency to be between 10 and 100 MHz for an atmospheric pressure capacitive discharge with a gap spacing of 0.1–0.4 cm. As for the reactive gas addition that is important for materials applications, we have been able to produce a stable discharge with up to 3%–4% of oxygen in helium for the 0.16 cm gap at 13.56 MHz. Even at this small fraction, the number density of oxygen molecules is high because of the atmospheric pressure operation, thus compensating this limitation. For example, we have previously shown that the oxygen atom density over 5×10^{15} cm^{-3} can be produced in the APPJ with 1% of oxygen[7] and this amount is sufficient to etch polyimide, tungsten, tantalum, and silicon dioxide at rates up to several μm/min, comparable to those achieved in low pressure discharge systems.[4,5] Still, further study is necessary to understand the gas breakdown and the formation of stable plasmas in a gas mixture at atmospheric pressure to reduce or replace the use of helium and to produce a wide range of chemistry in the APPJ.

In summary, gas breakdown phenomena were studied for a high pressure rf capacitive plasma source. At 13.56 MHz rf frequency, breakdown voltage is largely a function of the product of the pressure and the discharge gap spacing mostly independent of the gap spacing. However, breakdown voltages vary substantially with changes in rf frequency. These results have been successfully explained with a careful examination of the dominant electron loss mechanism using the HF breakdown theory by Brown. When argon, oxygen, or nitrogen is added to helium, a large increase in breakdown voltage was observed, which requires the use of helium and limits the maximum amount of reactive gases that can be added to helium feedgas. This limit on the gas composition may restrict the potential applications of this source and prompt the need for further study.

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